

**HIGHLY CONCENTRATED, STORAGE STABLE AQUEOUS DISPERSIONS FOR  
STABILISING COATINGS AND GLAZES**

5 Public opinion and legislation are increasingly imposing the requirement on the  
handlers of coating materials to lessen the emissions of solvents. The reasons given  
for these strictures are on the one hand the increased environmental burden and on  
the other hand the occupational hygiene situation. Aqueous coating materials have in  
recent years acquired a continually growing position in the market. Cataphoretic dip  
10 priming and the waterborne surfacer are now state of the art for the coating of  
automobiles. Water-based coatings have obtained line approval in recent years, and  
even water-based clearcoats are now already being used on the line. Even  
transportation sealing systems and protective waxes are now being applied on an  
aqueous basis. In the segments, too, of the coating of externally mounted components  
15 made of plastic or metal, the coating of agricultural and construction machinery, office  
furniture, electrical or electronic devices, shelving components, lamps, and so on,  
water-based coating materials are nowadays already being employed.

Water-based coating systems and glazes in which either water-soluble binders or  
20 water-dilutable binders, or a combination of water-soluble and water-dilutable binders,  
are used are generally mixable only with stabilizers that are inherently liquid. The  
majority of commercially available stabilizers, however, are solid, pulverulent, and of  
poor solubility, if not insoluble, in water.

25 Liquid products, solutions or dispersions additionally have a range of advantages over  
the powder forms, such as absence of dusting during handling, absence of wetting  
problems due to lumping, and, not least, ease of metering in automatic installations, for  
example. In the course of storage over prolonged periods, however, the durability of  
liquid preparations is often unsatisfactory, and accordingly the formation of  
30 agglomerates or of a sediment is frequently observed. Inadequately dispersed particles  
are often the cause of frequent blockages in automated metering and mixing systems.

All organic materials, whether natural or synthetic, must normally be protected against  
breakdown by environmental effects and UV light, through the addition of stabilizers, in  
35 particular through the addition of light stabilizers and antioxidants.

Aqueous dispersions of light stabilizers, particularly of UV absorbers, have been known for some time already. Their use is prior art primarily in connection with the dyeing of synthetic fibers, especially unmodified or acid-modified polyester fibers. The concentration of the active components that are present in the corresponding commercial forms is generally in the region of well below 40 percent by weight. In addition, it is frequently found that the storage stability of the dispersion is not adequate, as is manifested by sedimentation of the active substances.

EP 225287 describes dye bath preparations comprising as further adjuvants, for stabilizability, electrolyte-sensitive thickeners, in order to allow a defined viscosity range to be maintained.

EP 345219 describes aqueous dispersions of 2-(2'-hydroxyphenyl)benzotriazoles for improving the light fastness of dyeings on synthetic fibers. A required adjuvant is an acidic ester or its salt with an alkylene oxide adduct.

EP 354174 describes aqueous compositions of 2-(2'-hydroxyphenyl)benzotriazoles for improving the light fastness of dyeings on synthetic fiber materials, especially unmodified or acid-modified polyester fibers. The preparations include as a further component a condensation product of a non-ether-bridged aromatic sulfonic acid and formaldehyde.

EP 468921 describes the production of an aqueous preparation of o-hydroxyphenyl-triazines for improving the light fastness of dyeings on synthetic fiber materials, especially polyester fibers.

EP 474595 describes aqueous dispersions of specially substituted 2-(2'-hydroxyphenyl)benzotriazoles, alone or in a mixture with a benzophenone, for use in the dyeing of synthetic fiber materials, especially unmodified or acid-modified polyester fibers.

EP 490819 describes the aqueous preparation of a mixture of a benzotriazole compound and a 2-hydroxybenzophenone compound for use in the dyeing of synthetic fiber materials, especially unmodified or acid-modified polyester fibers.

EP 820978 describes the treatment of textile materials, to improve light stability, with

aqueous preparations of 2-hydroxybenzophenone compounds.

DE-A 3511924 describes aqueous preparations of sterically hindered amine light stabilizers (HALS) for use in the coatings field, said preparations containing generally  
5 5% to 50%, preferably 15% to 40% by weight of HALS and 2% to 30%, preferably 3% to 20% by weight of a nonionic dispersant. The active substance content used by way of example amounts to 32% by weight.

The dispersions prepared according to this laid-open specification, particularly those  
10 with relatively high active substance contents, however, are not sufficiently stable on storage to ensure a prolonged storage period without quality detractions.

GB 2 187 746 discloses stable aqueous dispersions of 2-(2'-hydroxyphenyl)benzo-triazoles (UV absorbers) having an active substance content of 20% to 45% by weight,  
15 and 7% to 15% by weight of a nonionic dispersant. The particle size of the UV absorber is preferably  $< 1\mu\text{m}$ . The active substance contents used by way of example amount to 40% by weight.

DE-A 19946519 discloses, very generally, aqueous dispersions for stabilizing  
20 polymeric dispersions, and also a process for preparing them. The active substance content (amount of phenolic antioxidant) is defined as being from 0.1% to 59.9% by weight, the fraction of surface-active substances for dispersing as being from 0.5% to 15% by weight.

25 The object of the present invention was to improve the storage stability of highly concentrated aqueous dispersions of a light stabilizer or of a mixture of a light stabilizer and an antioxidant.

The object is achieved in accordance with the invention by means of aqueous  
30 dispersions of a light stabilizer or of a mixture of a light stabilizer and an antioxidant, characterized in that they have an active substance content of more than 47% by weight and comprise at least one nonionic wetting agent as dispersant and a polyglycol as solubilizer, and also 0.2% to 5% by weight of oleic acid as flow improver.

35 The object is also achieved, further, by a method of stabilizing aqueous dispersions using the abovementioned components.

The present invention provides dispersions of individual or mixed light stabilizers and antioxidants and provides for their use in the stabilizing of coatings and glazes, especially water-based basecoats, clearcoats, and glazes. The invention relates in particular to storage stable aqueous preparations of solids that are of poor to zero solubility in water and are used as light stabilizers or antioxidants, with a particle size  $D_{50}$  of less than 5  $\mu\text{m}$ , for stabilizing water-based coating systems and glazes. The active substances preferably have a particle size of  $D_{50} = 0.5\text{--}2\ \mu\text{m}$  and  $D_{90} < 3.5\ \mu\text{m}$ .

10 The invention is characterized in that the highly concentrated, storage stable aqueous dispersions have an active substance content of more than 47% by weight, preferably 47%–57% by weight, more preferably 47%–54% by weight, and very preferably of more than 50% and up to 54% by weight.

15 The highly concentrated aqueous dispersions of the invention contain 0.2%–5.0% by weight of oleic acid as flow improver, to ensure an appropriate viscosity without excessive dilution. Preference is given to using amounts of 0.2%–4.0% by weight and with particular preference of 0.2%–3.0% by weight.

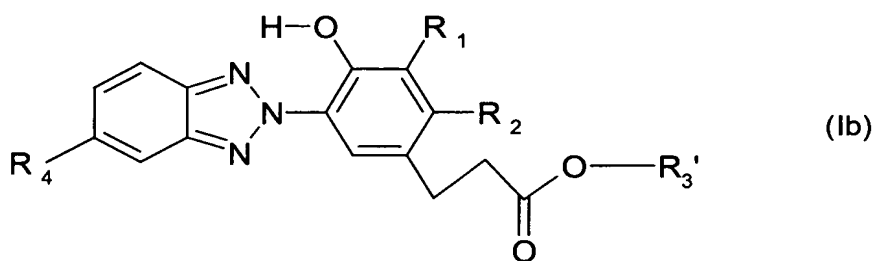
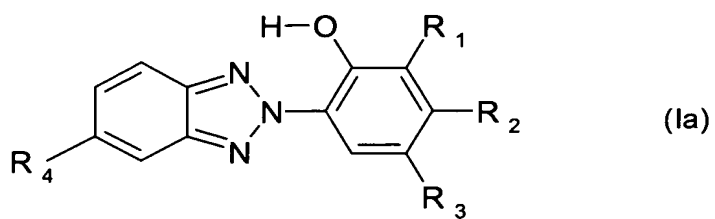
20 The aqueous dispersions preferably have a viscosity of 0.01 to 2 Pa s.

Besides nonionic wetting agent, the highly concentrated aqueous dispersions of the invention preferably also include an anionic wetting agent, which permits optimum dispersing of the active substances and thus ensures improved storage stability.

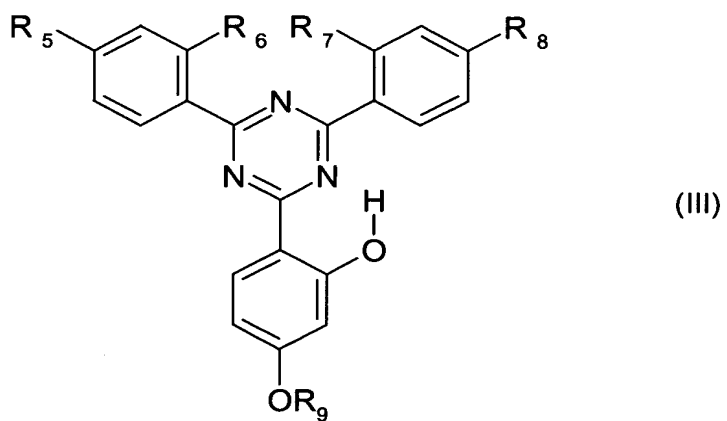
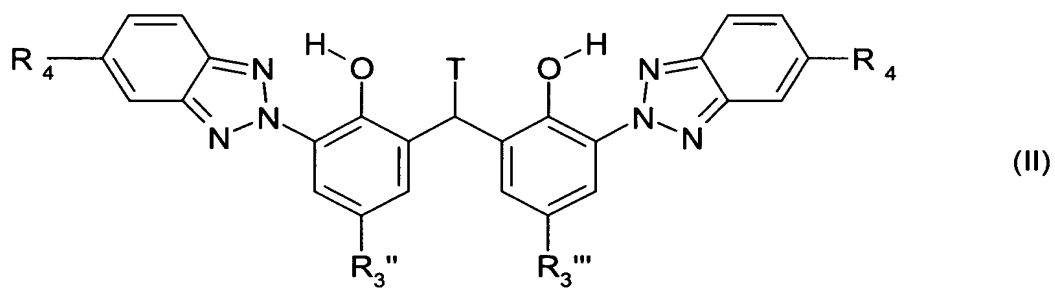
25 The light stabilizer, or the mixture of a light stabilizer and an antioxidant, in accordance with the invention, ought to have a melting point of at least 35°C in order that they do not melt in the course of processing to aqueous dispersions, which frequently take place at an elevated temperature.

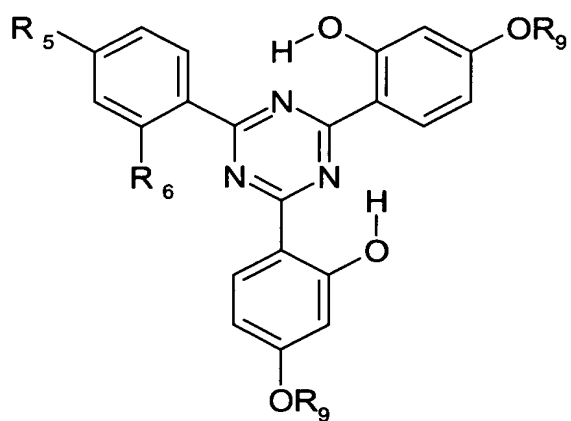
30 Preferred light stabilizers are the products with the formulae I to VIII

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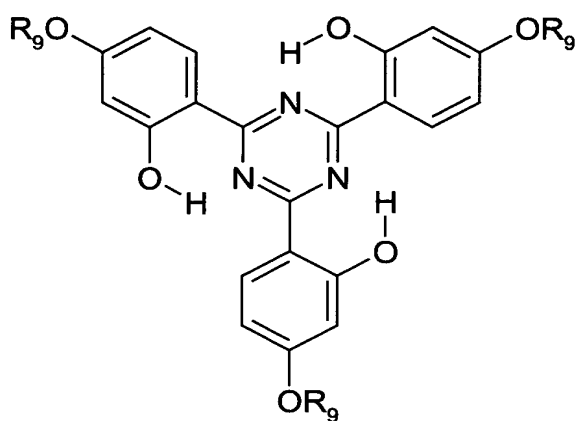


5 (solid products as described for example in US 4853471)

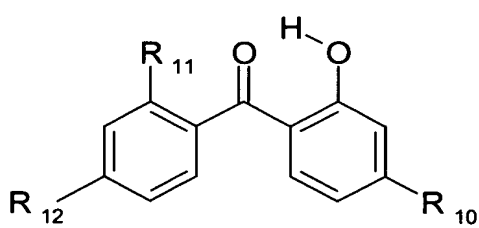




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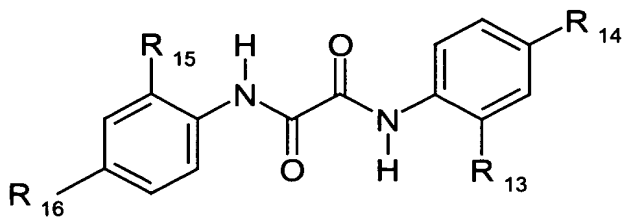


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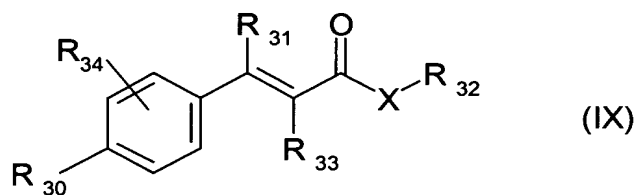
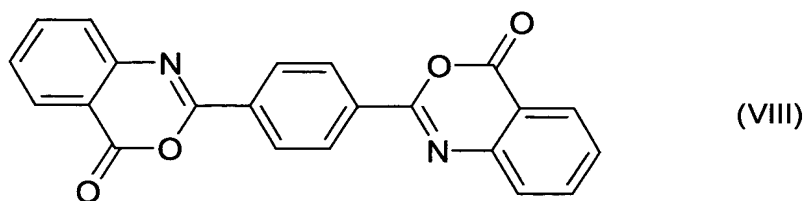


(VI)

(as described for example in EP 820 978 A1)

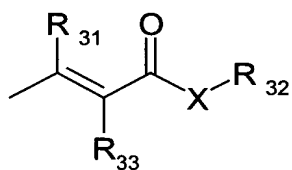


(VII)



5 in which

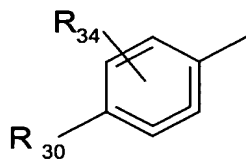
$R_{30}$  is H, n-alkyl, isoalkyl, unsubstituted or substituted aryl,  $OR_{35}$ , or a structural component



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where  $R_{35}$  is H, n-alkyl, isoalkyl, unsubstituted or substituted aryl,

$R_{31}$  is H, n-alkyl, isoalkyl or a structural component



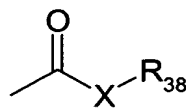
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X is -O- or  $-(NR_{36})-$

where  $R_{36}$  = H, n-alkyl, isoalkyl

20

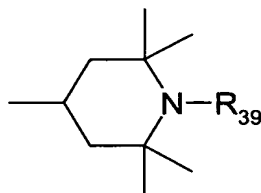
$R_{33}$  is H, n-alkyl, isoalkyl, CN or a group above, and



, where X being as described

$R_{32}$  and  $R_{38}$  independently have the following definitions:

H, n-alkyl, isoalkyl, unsubstituted and substituted aryl, or a structural component



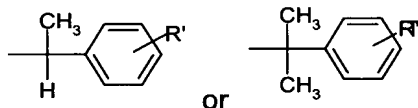
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where  $R_{39}$  = H or  $C_1$ - $C_{18}$  alkyl,  $C_7$ - $C_{18}$  alkylaryl, -O- $C_1$ - $C_{18}$  alkyl, or acetyl.

The symbols  $R_1$  to  $R_{16}$  have the following definitions:

10

$R_1$ ,  $R_2$ , and  $R_3$  independently of one another are hydrogen, linear or branched  $C_1$ - $C_{12}$  alkyl, cycloalkyl, aralkyl; preferably methyl, sec-butyl, tert-butyl, tert-amyl, tert-octyl, cyclohexyl or a group



15

in which  $R'$  is hydrogen or  $C_1$ - $C_{12}$  alkyl.

$R_4$  is hydrogen, halogen such as Cl or Br, carboxyl or a sulfonic acid group, linear or branched  $C_1$ - $C_{12}$  alkyl, cycloalkyl or lower alkoxy;

20

$R_3'$ ,  $R_3''$ , and  $R_3'''$  independently of one another are hydrogen, linear or branched  $C_1$ - $C_{12}$  alkyl, cycloalkyl, aralkyl;

$T$  is hydrogen,  $C_1$ - $C_{12}$  alkyl or an oxo group;

25

$R_5$ ,  $R_6$ ,  $R_7$ , and  $R_8$  independently of one another are hydrogen, linear or branched  $C_1$ - $C_8$  alkyl, unsubstituted aryl or lower alkyl-substituted aryl;

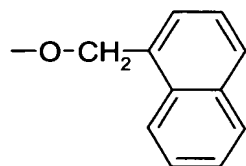
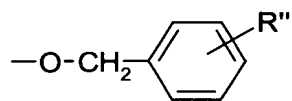
$R_9$  is hydrogen or a linear or branched, aliphatic or cycloaliphatic alkyl group with

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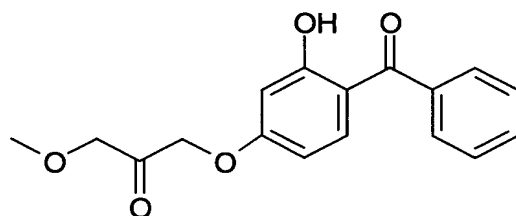
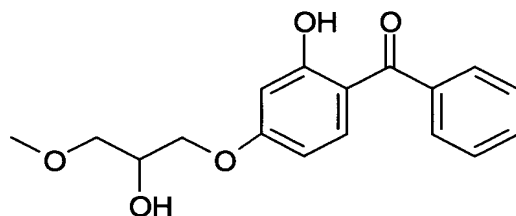
$C_1-C_{12}$  which optionally can be substituted by one or more OH groups or one or more alkoxy groups or mixedly by OH groups and alkoxy groups.

$R_{10}$  is OH, alkoxy with  $C_1-C_{12}$ , hydroxyethoxy or a group

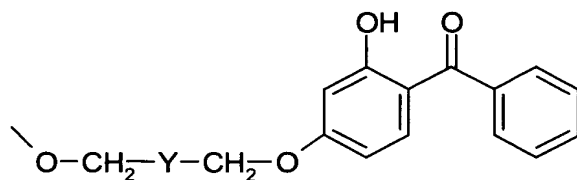
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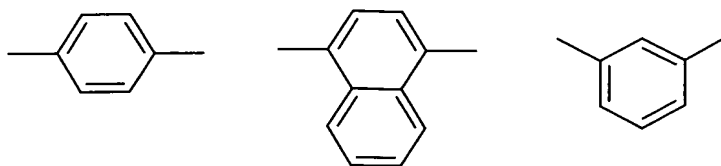
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in which Y is a direct bond or a group

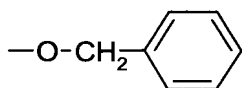


and in which R<sup>''</sup> is hydrogen, methyl or methoxy,

- 5 R<sub>11</sub> is hydrogen, halogen, cyano, C<sub>1</sub>-C<sub>8</sub> alkyl, cycloalkyl, alkoxy, phenoxy, phenyl, hydroxy or hydroxyethoxy;

R<sub>12</sub> is hydrogen, halogen, cyano, C<sub>1</sub>-C<sub>8</sub> alkyl, cycloalkyl, alkoxy, phenoxy, phenyl, hydroxy or hydroxyethoxy or a group

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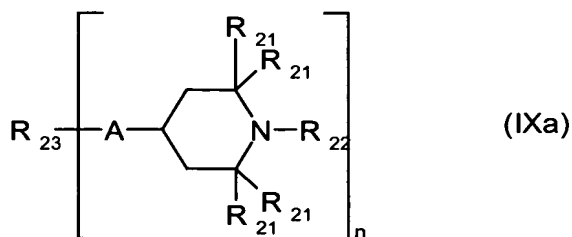


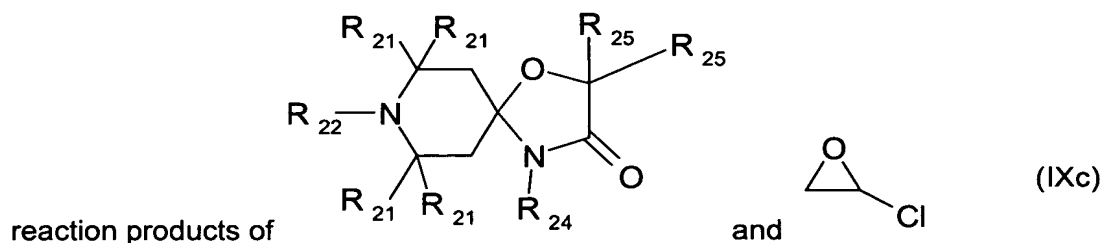
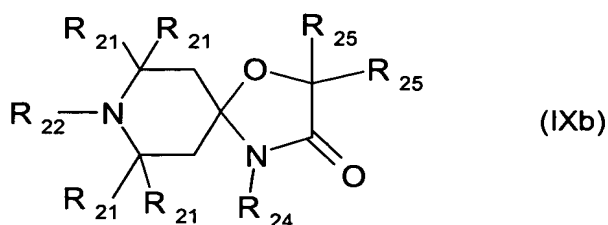
R<sub>13</sub>, R<sub>14</sub>, R<sub>15</sub>, and R<sub>16</sub>, identically or independently of one another, are hydrogen, linear or branched alkyl, OH or alkoxy.

15

The sterically hindered piperidine derivatives (HA(L)S) employed in accordance with the invention belong to the well-known group of stabilizers which are already used for improving light fastness or mechanical properties. Examples of sterically hindered piperidine derivatives of this kind are those of the formulae (IXa) - (IXc)

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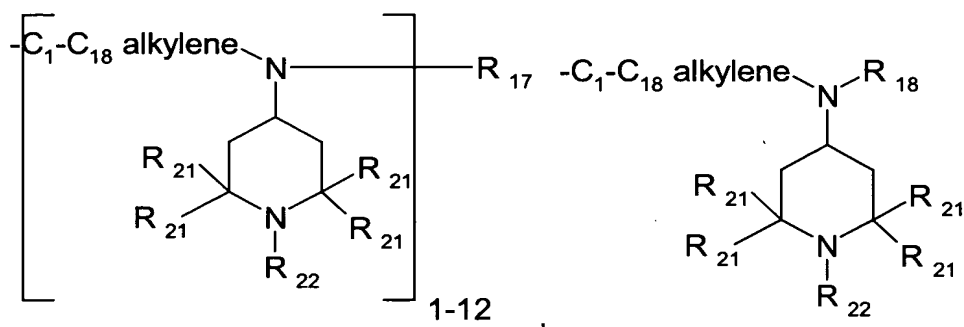




5 in which, independently of one another,

A is -O- or -NR<sub>20</sub>-,

R<sub>20</sub> is H, C<sub>1</sub>-C<sub>18</sub> alkyl or one of the groups



10

R<sub>17</sub> is H or C<sub>1</sub>-C<sub>18</sub> alkyl,

R<sub>18</sub> is H or an aliphatic, cycloaliphatic, aromatic or heteroaromatic radical,

R<sub>21</sub> is C<sub>1</sub>-C<sub>18</sub> alkyl,

15 or two radicals R<sub>21</sub> attached to the joint carbon atom are a C<sub>4</sub>-C<sub>8</sub> cycloalkyl radical,

R<sub>22</sub> is H or C<sub>1</sub>-C<sub>18</sub> alkyl, C<sub>7</sub>-C<sub>18</sub> alkylaryl,

R<sub>23</sub> is H or an n-valent aliphatic, cycloaliphatic, aromatic or heteroaromatic radical,

R<sub>24</sub> is C<sub>1</sub>-C<sub>18</sub> alkyl or -C(O)-C<sub>1</sub>-C<sub>18</sub> alkyl;

20 R<sub>25</sub> is H, C<sub>1</sub>-C<sub>18</sub> alkyl, C<sub>4</sub>-C<sub>18</sub> cycloalkyl, or two radicals R<sub>5</sub> attached to the same carbon atom are a C<sub>4</sub>-C<sub>18</sub> cycloalkyl radical,

n is an integer > 0.

As further additive the following antioxidants may be present in the dispersions of the invention:

5

- 1.1 alkylated monophenols, e.g., 2,6-di-tert-butyl-4-methylphenol, 2-butyl-4,6-dimethylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,6-di-tert-butyl-4-n-butylphenol, 2,6-di-tert-butyl-4-isobutylphenol, 2,6-dicyclopentyl-4-methylphenol, 2,4a-methylcyclohexyl-4,6-dimethylphenol, 2,6-dioctadecyl-4-methylphenol, 2,4,6-tricyclohexylphenol, 2,6-di-tert-butyl-4-methoxymethylphenol, 2,6-dinonyl-4-methylphenol, 2,4-dimethyl-6-(1'-methylundec-1'-yl)phenol, 2,4-dimethyl-6-(1'-methylheptadecyl)phenol, 2,4-dimethyl-6-(1'-methyltridec-1'-yl)phenol, and mixtures thereof.
- 1.2 alkylthiomethylphenols, e.g., 2,4-dioctylthiomethyl-6-tert-butylphenol, 2,4-dioctylthiomethyl-6-methylphenol, 2,4-dioctylthiomethyl-6-ethylphenol, 2,6-didodecylthiomethyl-4-nonylphenol.
- 1.3 hydroquinones and alkylated hydroquinones, e.g., 2,6-di-tert-butyl-4-methoxyphenol, 2,5-di-tert-butylhydroquinone, 2,5-di-tert-amylhydroquinone, 2,6-diphenyl-4-octadecyloxyphenol, 2,6-di-tert-butylhydroquinone, 2,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyphenyl stearate, bis(3,5-di-tert-butyl-4-hydroxyphenyl) adipate.
- 1.4 tocopherols, e.g.,  $\alpha$ -tocopherol,  $\beta$ -tocopherol,  $\gamma$ -tocopherol,  $\delta$ -tocopherol, and mixtures thereof (vitamin E).
- 1.5 hydroxylated thiodiphenyl ethers, e.g., 2,2'-thiobis(6-tert-butyl-4-methylphenol), 2,2'-thiobis(4-octylphenol), 4,4'-thiobis(6-tert-butyl-3-methylphenol), 4,4'-thiobis(6-tert-butyl-2-methylphenol), 4,4'-thiobis(3,6-di-sec-amylphenol), 4,4'-bis(2,6-dimethyl-4-hydroxyphenyl) disulfide.
- 1.6 alkylidene bisphenols, e.g., 2,2'-methylenebis(6-tert-butyl-4-methylphenol), 2,2'-methylenebis(6-tert-butyl-4-ethylphenol), 2,2'-methylenebis[4-methyl-6-( $\alpha$ -methylcyclohexyl)phenol], 2,2'-methylenebis(4-methyl-6-cyclohexylphenol), 2,2'-methylenebis(6-nonyl-4-methylphenol), 2,2'-methylenebis(4,6-di-tert-butylphenol),

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- 2,2'-ethylidenebis(4,6-di-tert-butylphenol), 2,2'-ethylidenebis(6-tert-butyl-4-isobutylphenol), 2,2'-methylenebis[6-( $\alpha$ -methylbenzyl)-4-nonylphenol], 2,2'-methylenebis[6-( $\alpha,\alpha$ -dimethylbenzyl)-4-nonylphenol], 4,4'-methylenebis(2,6-di-tert-butylphenol), 4,4'-methylenebis(6-tert-butyl-1-2-methylphenol), 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 2,6-bis(3-tert-butyl-5-methyl-2-hydroxybenzyl)-4-methylphenol, 1,1,3-tris(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-3-n-dodecylmercaptobutane, ethylene glycol bis[3,3-bis(3'-tert-butyl-4'-hydroxyphenyl)-butyrate], bis(3-tert-butyl-4-hydroxy-5-methylphenyl)dicyclopentadiene, bis[2-(3'-tert-butyl-2'-hydroxy-5'-methylbenzyl)-6-tert-butyl-4-methylphenyl] terephthalate, 1,1-bis(3,5-dimethyl-2-hydroxyphenyl)butane, 2,2-bis(3,5-di-tert-butyl-4-hydroxyphenyl)propane, 2,2-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-4-n-dodecylmercaptobutane, 1,1,5,5-tetra(5-tert-butyl-4-hydroxy-2-methylphenyl)-pentane.
- 1.7 O-, N-, and S-benzyl compounds, e.g., 3,5,3',5'-tetra-tert-butyl-4,4'-dihydroxy-dibenzyl ether, octadecyl 4-hydroxy-3,5-dimethylbenzylmercaptoacetate, tris(3,5-di-tert-butyl-4-hydroxybenzyl)amine, bis(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl) dithioterephthalate, bis(3,5-di-tert-butyl-4-hydroxybenzyl) sulfide, isooctyl 3,5-di-tert-butyl-4-hydroxybenzylmercaptoacetate.
- 1.8 hydroxybenzylated malonates, e.g., dioctadecyl 2,2-bis(3,5-di-tert-butyl-2-hydroxybenzyl)malonate, dioctadecyl 2-(3-tert-butyl-4-hydroxy-5-methylbenzyl)-malonate, didodecyl mercaptoethyl-2,2-bis(3,5-di-tert-butyl-4-hydroxybenzyl)-malonate, di[4-(1,1,3,3-tetramethylbutyl)phenyl] 2,2-bis(3,5-di-tert-butyl-4-hydroxybenzyl)malonate.
- 1.9 aromatic hydroxybenzyl compounds, e.g., 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-2,4,6-trimethylbenzene, 1,4-bis(3,5-di-tert-butyl-4-hydroxybenzyl)-2,3,5,6-tetramethylbenzene, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)phenol.
- 1.10 triazine compounds, e.g., 2,4-bis(octylmercapto-6-(3,5-di-tert-butyl-4-hydroxyanilino))-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyanilino)-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,3,5-triazine, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,2,3-triazine, 1,3,5-

- 5 tris(3,5-di-tert-butyl-4-hydroxybenzyl)isocyanurate, 1,3,5-tris(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl)isocyanurate, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenylethyl)-1,3,5-triazine, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hexahydro-1,3,5-triazine, 1,3,5-tris(3,5-dicyclohexyl-4-hydroxybenzyl)isocyanurate.
- 10 1.11 benzylphosphonates, e.g., dimethyl 2,5-di-tert-butyl-4-hydroxybenzylphosphonate, diethyl 3,5-di-tert-butyl-4-hydroxybenzylphosphonate, dioctadecyl 3,5-di-tert-butyl-4-hydroxybenzyl phosphonate, dioctadecyl 5-tert-butyl-4-hydroxy-3-methylbenzylphosphonate, Ca salt of monoethyl 3,5-di-tert-butyl-4-hydroxybenzylphosphonate.
- 15 1.12 acylaminophenols, 4-hydroxylauramide, 4-hydroxystearanilide, octyl N-(3,5-di-tert-butyl-4-hydroxyphenyl)carbamate.
- 20 1.13 esters of  $\beta$ -(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with mono- or polyhydric alcohols, e.g., with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl)isocyanurate, N,N'-bis(hydroxyethyl)oxalamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.
- 25 1.14 esters of  $\beta$ -(5-tert-butyl-4-hydroxy-3-methylphenyl)propionic acid with mono- or polyhydric alcohols, e.g., with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxy)ethylisocyanurate, N,N'-bis(hydroxyethyl)oxalamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.
- 30 1.15 esters of  $\beta$ -(3,5-dicyclohexyl-4-hydroxyphenyl)propionic acid with mono- or polyhydric alcohols, e.g., with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxy)ethylisocyanurate, N,N'-bis(hydroxyethyl)oxalamide,
- 35

3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

- 1.16 esters of 3,5-di-tert-butyl-4-hydroxyphenylacetic acid with mono- or polyhydric  
 5 alcohols, e.g., with methanol, ethanol, octanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxy)ethylisocyanurate, N,N'-bis(hydroxyethyl)oxalamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-  
 10 hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.
- 1.17 amides of  $\beta$ -(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, such as N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hexamethylenediamine, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)trimethylenediamine, N,N'-bis(3,5-  
 15 di-tert-butyl-4-hydroxyphenylpropionyl)hydrazine.

Further additives which may be present in the dispersions of the invention are as follows:

- 20 2. metal deactivators, such as N,N'-diphenyloxalamide, N-salicylal-N'-salicyloylhydrazine, N,N'-bis(salicyloyl)hydrazine, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hydrazine, 3-salicyloylamino-1,2,4-triazole, bis(benzylidene)oxalyl dihydrazide, oxanilide, isophthaloyl dihydrazide, sebacoyl bisphenylhydrazide, N,N'-diacetyl adipoyl dihydrazide, N,N'-bissalicyloyloxalyl  
 25 dihydrazide, N,N'-bissalicyloylthiopropionyl dihydrazide.
3. phosphites and phosphonites, such as triphenyl phosphite, diphenylalkyl phosphites, phenyl dialkyl phosphites, tris(nonylphenyl) phosphite, trilauryl phosphite, trioctadecyl phosphite, distearyl pentaerythritol diphosphite, tris(2,4-di-  
 30 tert-butylphenyl) phosphite, diisodecyl pentaerythritol diphosphite, bis(2,4-di-tert-butylphenyl) pentaerythritol diphosphite, bis(2,6-di-tert-butyl-4-methylphenyl) pentaerythritol diphosphite, bis-isodecyloxy-pentaerythritol diphosphite, bis(2,4-di-tert-butyl-6-methylphenyl) pentaerythritol diphosphite, bis(2,4,6-tri-tert-butylphenyl) pentaerythritol diphosphite, tristearylsorbitol triphosphite,  
 35 tetrakis(2,4-di-tert-butylphenyl) 4,4'-biphenylene diphosphonite, 6-isooctyloxy-2,4,8,10-tetra-tert-butyl-12H-dibenzo[d,g]-1,3,2-dioxaphosphocine, 6-fluoro-

2,4,8,10-tetra-tert-butyl-12-methyldibenzo[d,g]-1,3,2-dioxaphosphocine, bis(2,4-di-tert-butyl-6-methylphenyl) methyl phosphite, bis(2,4-di-tert-butyl-6-methylphenyl) ethyl phosphite.

- 5     4.    peroxide scavenger compounds, such as esters of  $\beta$ -thiodipropionic acid, examples being the lauryl, stearyl, myristyl or tridecyl esters, mercaptobenzimidazole, the zinc salt of 2-mercaptobenzimidazole, zinc dibutyldithiocarbamate, dioctadecyl disulfide, pentaerythritol tetrakis( $\beta$ -dodecylmercapto)propionate.
- 10    5.    polyamide stabilizers, such as copper salts in combination with iodides and/or phosphorus compounds, and salts of divalent manganese.
- 15    6.    basic co-stabilizers, such as melamine, polyvinylpyrrolidone, dicyandiamide, triallyl cyanurate, urea derivatives, hydrazine derivatives, amines, polyamides, polyurethanes, alkali metal salts and alkaline earth metal salts of higher fatty acids, examples being Ca stearate, Zn stearate, Mg behenate, Mg stearate, Na ricinoleate, K palmitate, antimony pyrocatecholate or tin pyrocatecholate.
- 20    7.    nucleating agents, such as 4-tert-butylbenzoic acid, adipic acid, diphenyl acetic acid.
- 25    8.    fillers and reinforcing agents, such as calcium carbonate, silicates, glass fibers, asbestos, talc, kaolin, mica, barium sulfate, metal oxides, metal hydroxides, carbon black, graphite.
- 30    9.    other adjuvants, such as plasticizers, lubricants, emulsifiers, pigments, optical brighteners, flame retardants, antistats, blowing agents.

30    The highly concentrated aqueous dispersions of the invention have a storage stability of more than 4 weeks at 50°C, which experience indicates corresponds to a storage of more than 1 year at room temperature.

35    In one preferred embodiment the aqueous dispersions of the invention include a biocide as a further component. The presence of a biocide further considerably increases the storage stability, since the decomposition (or partial degradation) of the dispersant and/or of the light stabilizers through fungal and/or bacterial infestation is

prevented or at least greatly slowed.

Biocides are, for example, compounds from the class of the derivatized isothiazolin-3-ones. They are used in the form of their solutions, emulsions, or as synergistic mixtures with other components. Preference is given to commercially customary biocides such as, for example, the commercial products ACTICIDE® from Thor or PARMETOL® from Schülke & Mayr.

Particularly preferred aqueous dispersions in accordance with the invention contain 47%-54% by weight active substance, 5%-10% by weight wetting agents, 5%-10% by weight solubilizers, 0.2%-3% by weight oleic acid, and < 1% by weight biocides, in 30%-40% by weight water.

The stabilizer preparations of the invention can be prepared by dispersing the stabilizer in the form of a powder, compact or granules in the presence of the oleic acid, the polyglycol and dispersant and/or further customary additives. The dispersant and/or additives are preferably first mixed with the oleic acid, the stabilizer is incorporated with stirring, and, depending on the harshness of the particulate nature of the stabilizer employed, the mixture is dispersed using agitator mechanisms, dissolvers, rotor-stator mills, bore mills, agitator-mechanism bore mills, such as sand mills and bead mills, high-speed mixers, kneading apparatus or high-power bead mills. The preparation obtained is optionally diluted further with oleic acid.

The highly concentrated aqueous dispersions of the invention are incorporated into aqueous coating compositions, where they are used in amounts of 0.3% to 3% by weight of the overall formula and are compatible with all known binder systems. Examples of such systems are aqueous dispersions, aqueous emulsions or aqueous solutions of a binder based on crosslinkable alkyd resin, acrylic resin, polyester resin or polyurethane resin.

30

The examples below use the following products:

UVA 1 = Tinuvin® 326	2-(2'-hydroxy-3'-tert-butyl-5'-methylphenyl)-5-chlorobenzotriazole
UVA 2 = Tinuvin® P	2-(2'-hydroxy-5'-methylphenyl)benzotriazole
UVA 3 = Tinuvin® 328	2-(2'-hydroxy-3',5'-di-tert-amylphenyl)benzotriazole

	UVA 4 = Tinuvin® 329	bis-[2-hydroxy-5-tert-octyl-3-(benzotriazol-2-yl)-phenyl]methane
	UVA 5 = Cyasorb® 1164 P	2-[4,6-bis(2,4-dimethylphenyl)-1,3,5-triazin-2-yl]-5-(octyloxy)phenol
5	UVA 6 = Tinuvin® 405	2-[4,6-bis(2,4-dimethylphenyl)-1,3,5-triazin-2-yl]-5-[3-[(2-ethylhexyl)oxy]-2-hydroxypropoxy]phenol
	UVA 7 = Tinuvin® 1577	2-(4,6-diphenyl-1,3,5-triazin-2-yl)-5-hexyloxyphenol
	UVA 8 = Sanduvor® 3041	2-hydroxy-4-methoxybenzophenone
	UVA 9 = Uvinul® 3040	2,2'-dihydroxy-4-methoxybenzophenone
10	UVA 10 = Sanduvor® 3035	2-hydroxy-4-n-octyloxybenzophenone
	UVA 11 = Sanduvor® VSU	N-(2-ethoxyphenyl)-N'-(2-ethylphenyl)ethanediamide
	UVA 12 = Sanduvor® EPU	N-(2-ethoxy-5-tert-butylphenyl)-N'-(2-ethylphenyl)-ethanediamide
	UVA 13 = Cyasorb® UV-3638	2,2'-(1,4-phenylene)bis[4H-3,1-benzoxazin-4-one]
15	UVA 14	methyl cinnamate
	UVA 15 = Uvinul® 3035	ethyl 2-cyano-3,3-diphenylacrylate
	UVA 16 = Hostavin® B-CAP	tetraethyl 2,2'-bis(1,4-phenylenedimethylidene)-malonate
20	UVA 17 = Cyasorb® UV-3346	poly[(6-morpholino-s-triazine-2,4-diyl)[2,2,6,6-tetramethyl-4-piperidyl]imino]hexamethylene[(2,2,6,6-tetramethyl-4-piperidyl)imino]]
	HALS 1 = Tinuvin® 770	bis(2,2,6,6-tetramethyl-4-piperidyl) sebacate
	HALS 2 = Tinuvin® 144	bis(1,2,2,6,6-pentamethyl-4-piperidyl) (3,5-di-tert-butyl-4-hydroxybenzyl)butylpropanedioate
25	HALS 3 = Sanduvor® PR-31	propanedioic acid, [(4-methoxyphenyl)methylene]-bis(1,2,2,6,6-pentamethyl-4-piperidyl) ester
	HALS 4 = Sanduvor® 3051	2,2,4,4-tetramethyl-7-oxa-3,20-diazadispiro-[5.1.11.2]heneicosan-21-one
30	HALS 5 = Hostavin® N30	polymer from the reaction of 2,2,4,4-tetramethyl-7-oxa-3,20-diazadispiro{5.1.11.2}heneicosan-21-one with epichlorohydrin
	HALS 6 = Chimassorb® 944	poly[(6-isooctylamino-s-triazine-2,4-diyl)[2,2,6,6-tetramethyl-4-piperidyl]imino]hexamethylene-[(2,2,6,6-tetramethyl-4-piperidyl)imino]]
35	HALS 7 = Chimassorb® 119	1,3,5-triazine-2,4,6-triamine, N,N'''-[1,2-ethanediy]-

		bis[[[4,6-bis[butyl(1,2,2,6,6-pentamethyl-4-piperidiny]amino]-1,3,5-triazin-2-yl]imino]-3,1-propanediyl]]bis[N',N''-dibutyl-N',N''-bis(1,2,2,6,6-pentamethyl-4-piperidiny]
5	HALS 8	4-hydroxy-2,2,6,6-tetramethylpiperidiny-1-oxy
	Dispersant mixture M1	oleyl-8-ethoxylate + tetrasodium N-(1,2-dicarboxyethyl)-N-octadecylsulfosuccinamate
10	Dispersant mixture M2	oleyl-10-ethoxylate + tetrasodium N-(1,2-dicarboxyethyl)-N-octadecylsulfosuccinamate

The polyglycol used is a polyethylene glycol monomethyl ether having an average mass in the range 470-530 g/mol.

15

**EXAMPLE 1**

In a vertical mill

- 10 parts of polyglycol
- 8.5 parts of a dispersant mixture M1
- 20 1.0 part of oleic acid and
- 0.2 part of a biocide are mixed in 32.3 parts of water.

Then 48 parts of UVA 1 are added and the mixture is ground with glass beads (diameter 1 mm) for 5-6 hours until the particle size is < 5 µm. The dispersion thus obtained is separated from the beads by filtration. This gives a storage stable dispersion (viscosity 1.0 Pas).

25

**EXAMPLE 2**

By the same process,

- 30 20 parts of polyglycol
- 6 parts of dispersant mixture M2
- 1.0 part of oleic acid and
- 0.2 part of a biocide are mixed in 24.8 parts of water, and 48 parts of UVA 1 are added. This gives a storage stable dispersion (viscosity 1.8 Pas).

35

**EXAMPLE 3**

By the same process,

- 5 parts of polyglycol
  - 6 parts of dispersant mixture M2
  - 5 1.0 part of oleic acid and
  - 0.2 part of a biocide are mixed in 39.8 parts of water, and 48 parts of UVA 1
- are added. This gives a storage stable dispersion (viscosity 0.8 Pas).

10 **EXAMPLE 4**

By the same process,

- 10 parts of polyglycol
  - 6 parts of dispersant mixture M2
  - 1.0 part of oleic acid and
  - 15 0.2 part of a biocide are mixed in 30.8 parts of water, and 52 parts of UVA 2
- are added. This gives a storage stable dispersion (viscosity 0.7 Pas).

**EXAMPLE 5**

20 By the same process,

- 5 parts of polyglycol
  - 6 parts of dispersant mixture M2
  - 1.0 part of oleic acid and
  - 0.2 part of a biocide are mixed in 39.8 parts of water, and 48 parts of UVA 3
- 25 are added. This gives a storage stable dispersion (viscosity 0.2 Pas).

**EXAMPLE 6**

By the same process,

- 30 5 parts of polyglycol
  - 6 parts of dispersant mixture M2
  - 1.0 part of oleic acid and
  - 0.2 part of a biocide are mixed in 37.8 parts of water, and 50 parts of UVA 4
- are added. This gives a storage stable dispersion (viscosity 0.4 Pas).

35

**EXAMPLES 7 - 28**

By the same process, in accordance with Example 3 or 5 respectively,

- 5 parts of polyglycol
- 6 parts of dispersant mixture M2
- 5 1.0 part of oleic acid and
- 0.2 part of a biocide

are mixed in 37.8 parts of water, and 48 parts of the following stabilizers are added:

- Example 7 UVA 5
- 10 Example 8 UVA 6
- Example 9 UVA 7
- Example 10 UVA 8
- Example 11 UVA 9
- Example 12 UVA 10
- 15 Example 13 UVA 11
- Example 14 UVA 12
- Example 15 UVA 13
- Example 16 UVA 14
- Example 17 UVA 15
- 20 Example 18 UVA 16
- Example 19 HALS 1
- Example 20 HALS 2
- Example 21 HALS 3
- Example 22 HALS 4
- 25 Example 23 HALS 5
- Example 24 HALS 6
- Example 25 HALS 7
- Example 26 UVA 17
- Example 27 HALS 4 + HALS 8
- 30 Example 28 HALS 4 + UVA 11

**DEMONSTRATION OF IMPROVED STORAGE STABILITY**

- A sample of each of the dispersions prepared according to DE-A 3511924 Example 1
- 35 and Example 2 is tested with the dispersions according to the above-described
- Examples 22 and 27, respectively, in respect of their storage stabilities at room

temperature and 50°C. The measure taken for the stability is the time after which the products begin to settle:

	Storage at room temperature	Storage at 50°C
DE-A 3511924 Example 1 (active substance content 32% by weight)	8-9 weeks	6 weeks
Example 22 (active substance content 48% by weight)	22 weeks	18-19 weeks
DE-A 3511924 Example 2 (active substance content 32% by weight)	12 weeks	8-9 weeks
Example 27 (active substance content 48% by weight)	24 weeks	20 weeks

5

#### **DEMONSTRATION OF IMPROVED STORAGE STABILITY THROUGH ADDITION OF A BIOCIDES**

Examples 1 to 6 are repeated without a biocide in the formula (Examples 1a to 6a).

- 10 After 6 months' storage at room temperature the specimens are investigated for fungal and bacterial infestation.

The test system used is from the company Biotest Diagnostics GK-T/HS, allowing simultaneous determination of the total microbial count/TTC on one side and of the yeasts and molds on the other side. For this purpose, the dispersions are applied slowly dropwise over both sides of the indicator surfaces, so that the surfaces are wetted, or they are brushed with a sterile inoculation loop immersed in the dispersions. The tubes are subsequently sealed and incubated in an incubator at 30-35°C for 3-5 days. In accordance with the instructions accompanying each pack, the indicators are evaluated by comparison. If after the appropriate incubation time there are more than 10<sup>2</sup> CFU/ml (CFU = colony forming units) present, then there is severe contamination of the dispersion, which may have a strongly negative influence on the further storage stability of the dispersion (degradation of the dispersant).

20

With biocide	CFU	Without biocide	CFU
Example 1	$< 10^2$	Example 1a	$\geq 10^5$
Example 2	$< 10^2$	Example 2a	$> 10^4$
Example 3	$< 10^2$	Example 3a	$> 10^3$
Example 4	$< 10^2$	Example 4a	$> 10^3$
Example 5	$< 10^2$	Example 5a	$> 10^4$
Example 6	$< 10^2$	Example 6a	$> 10^3$

### **APPLICATION EXAMPLE**

- 5 A water-based resin system consisting of a styrene-modified acrylate emulsion is prepared as follows: a mixture consisting of

106.0 parts of butylglycol and

132.1 parts of demineralized water

is added with thorough stirring and slowly to

- 10 761.9 parts of Neocryl® XK-62 (Avecia).

This system serves in the further experiments as base material for testing the dispersions cited in the examples in respect, for example, of clear solubility, which is an indicator of good compatibility and distribution.

15

The "coating materials" stabilized in this way are applied using a doctor blade (film width 80 mm) at 100 µm wet film thickness to highly transparent polyester films and/or matt black-pigmented LENETA charts, and dried in a forced-air oven at 50°C and 80°C. Thereafter the polyester films are inspected for transparency, the LENETA charts for any blooming, which represents an indicator of incompatibility and saturation in the coating material.

20

### **APPLICATION IN AN AQUEOUS 2K [2-COMPONENT] PU CLEARCOAT FOR OEM**

#### **APPLICATIONS**

The polyol component is put together on the basis of the following ingredients:

Bayhydrol® VP LS 2271 28.33 (Bayer)

Bayhydrol® VP LS 2231 31.03 (Bayer)

Byk® 345 as-supplied form 0.29 (Byk Chemie)

Byk® 333 25% in water	0.28	(Byk Chemie)
Distilled water	12.86	

- 5 The polyol component is stirred up intensively by hand or using a high-speed stirrer (1000 rpm). The individual ingredients should be mixed in the predetermined order.

### **CURING COMPONENT**

	Desmodur® N 3600	17.28	(Bayer)
10	Butylglycol acetate/Solvesso® 100 (1 : 1)	7.33	(Exxon Mobile)

- 15 Prior to mixing with the curing component, the dispersions described in the examples are added to the polyol component. Mixing with the curing component should not take place any earlier than 12 hours after the preparation of the polyol component. Mixing of the polyol component with the curing component is accomplished in 2 steps:

- 1) by preparing a preliminary emulsion by stirring together with components by hand or
- 2) laboratory stirrer

- 20 Final mixing is accomplished by jet dispersion at 50 bar (e.g., airless pump Merkur 060.020-DP F, Walther, Wuppertal) or using a Dispermate (5 min at 2000 rpm).

Comparative experiments by addition of liquid HALS stabilizers based on butylglycol to the resin solution:

25	Tinuvin® 292 (Ciba)	severe gelling after a short time
	Sanduvor® 3052 (Clariant)	severe gelling after a short time

### **APPLICATION IN A SOFT-FEEL COATING**

- 30 The soft-feel coating material is prepared as follows:

Item	Raw material	Amount	Description
1	CUR 21	54.70	Binder (Alberdingk-Boley)
2	Byk 024	0.60	Defoamer (Byk Chemie)
3	Dow Corning 67	0.40	Wetting agent (Dow Corning)

4	Byk 044	0.20	Defoamer (Byk Chemie)
5	Worlée-Pol 1181/03	5.00	Polyester (Worlée)
6a	Butylglycol*	3.50	Solvent (BASF)
6b	Water*	30.00	Solvent
7	Acematt TS-100	1.50	Matting agent (Degussa)
8	DSX 1514	0.10	Rheology additive (Cognis)
9	Colanyl black N230	4.00	Pigment preparation (Clariant)
		100.00	

\* = Mix before use!

Added to this coating material is 0.5% by weight of the above-described dispersion.

- 5 The "coating materials" thus stabilized are applied using a doctor blade (film width 80 mm) at a wet film thickness of 100  $\mu$ m to highly transparent polyester films, and dried at room temperature and at 50°C in a forced-air oven. Thereafter the polyester films are tested for transparency.